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(54) DISPERSANT AND INORGANIC FINE PARTICLE DISPERSION USING THE SAME

(57)Abstract:

PURPOSE: To obtain a dispersant having good dispersing function and improved in the compatibility with an org. component by using a polymer containing α,β -monoethylenic unsaturated carboxylic acid or a salt thereof as a principal constituent monomer as the dispersant dispersing fine particles in alkylene glycol.

CONSTITUTION: A polymer containing α,β -monoethylenic unsaturated carboxylic acid or a salt thereof as a principal constituent monomer is used as a dispersant dispersing fine particles of titanium dioxide or calcium carbonate in alkylene glycol such as ethylene glycol. The ratio of α,β -monoethylenic unsaturated carboxylic acid or a salt thereof is set to 20% or more, pref., 40% or more by wt. of all of monomers. This dispersant well disperses fine particles of pigment or the like.

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CLAIMS

[Claim(s)]

[Claim 1]A dispersing agent which dissolves a polymer which makes alpha, beta mono- ethylenic unsaturated carboxylic acid, or its salt a main composition monomer in a solvent containing alkylene glycol.

[Claim 2]The dispersing agent according to claim 1 whose rate of closing into alpha, beta mono- ethylenic unsaturated carboxylic acid, or all the composition monomers of the salt is 20 % of the weight or more.

[Claim 3]The dispersing agent according to claim 1 whose salts of alpha and beta mono- ethylenic unsaturated carboxylic acid are at least one sort of salts chosen from alkali metal salt, ammonium salt, and amine salt.

[Claim 4]The dispersing agent according to claim 1 whose content of alkylene glycol in a solvent is 150 or less weight section more than per weight section per polymer 100 weight section.

[Claim 5]The dispersing agent according to claim 1 which is a copolymer in which the polymer according to claim 1 also makes a composition monomer polyalkylene glycol monoacrylate or mono- methacrylate.

[Claim 6]Inorganic particulate dispersion liquid which it comes to distribute with the dispersing agent according to claim 1.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application]About a dispersing agent, in more detail, when this invention distributes inorganic particles (it is only hereafter described as particles in many cases), such as paints, in alkylene glycol, such as ethylene glycol, it relates to the particulate dispersion liquid which it comes to distribute with a suitable dispersing agent and this dispersing agent.

[0002]

[Description of the Prior Art]Now, particles, such as particles especially a titanium dioxide, barium sulfate, calcium carbonate, talc, and kaolin, are widely used in large quantities as a loading material or paints, such as rubber, a plastic, paper, and a paint. For example, in the paper-making industry, although a lot of calcium carbonate is used with talc and kaolin, In order to think the surface gloss of coated paper as important, good calcium carbonate of dispersibility is demanded, Distribution underwater [with many aggregate particles] changes to the granular material of difficult calcium carbonate, and it is used in a dispersion-liquid figure, supplying the high concentration drainage system dispersing element which the calcium carbonate maker prepared to a paper manufacturer. Since the dispersing agent in which the solution of sodium polyacrylate of the high concentration drainage system dispersion liquid of this calcium carbonate, etc. are good is marketed, that preparation is easy.

[0003]The particles prepared especially very with careful attention to the homogeneity of particle diameter and dispersibility among the above mentioned particles, For example, synthetic silica prepared by hydrolyzing alkoxysilane etc., calcium carbonate which grinds the synthetic calcium carbonate which has a specific degree of dispersion under a specific condition, and is prepared, Particles, such as kaolin which repeat advanced centrifugal classification and is prepared, are used as an antiblocking agent of the polyester film used as materials, such as magnetic tape, such as an audiotape and videotape. In order that these inorganic compounds may improve dispersibility in the inside of polyester, prepare the glycol dispersion liquid of particles and adding to the manufacturing process of polyester is performed, but. It cannot say that these particles have good dispersibility and dispersion stability in the inside of glycol, but when long term storage of the glycol which made particles suspended is carried out, particles carry out sedimentation precipitation and form a hard hard cake, and re dispersion becomes difficult, There is also a fault that furthermore an inorganic compound will condense at the time of manufacture of the inside of glycol or polyester. When a condensation coarse particle exists in polymer, become a cause of the thread breakage at the time of the spinning of polyester fiber, or, In becoming causes, such as a big and rough projection and a fish eye, in a film, especially using it for the film for magnetic tape, in order to cause a dropout and the fall of a S/N ratio, it waits for development of the dispersion techniques of the particles which a condensation coarse particle does not generate.

[0004]In polyester film, the slide nature and *****-proof are a quality of the workability of the manufacturing process of a film, and the work process in each use, and a major factor which influences the quality of the product quality further. In applying a magnetic layer to the polyester film surface, for example when these slide nature and *****-proof are insufficient, and using as magnetic tape, Friction with the coating roll and film surface at the time of magnetic layer spreading is intense, and wear of the film surface by this is also intense, and when extreme, the wrinkles to a film surface, an abrasion, etc. occur. Also after carrying out the slit of the film after magnetic layer spreading and processing it into an audio, video, or the tape for computers, In the case of operation of the cash drawer from a reel, a cassette, etc., and winding up and others, many guide parts, wear arises remarkably between playback heads etc. -- an abrasion and distorted generating -- the polyester film surface can be deleted further -- etc. -- as a result of

depositing the face powder-like substance to twist, it becomes a big cause of lack of a magnetic-recording signal, i.e., a dropout, in many cases.

[0005] Since this does not have the enough compatibility of polyester which is particles and an organic component, exfoliation occurs on the boundary of particles and polyester at the time of extension, etc., and a void generates it. When this void exists in polyester, by contact with a polyester film comrade or polyester film, and other substrates. Particles become being easy to be desorbed by damage to polyester film, etc. from polyester film generating of face powder and the cause of a dropout in the film for magnetic tape as mentioned above. Since a big opening exists around a particle, it comes to spoil the transparency of polyester film. For this reason, it is supposed that it is lack of the compatibility of particles and polyester a problem which should be solved in respect of abrasion resistance and transparency.

[0006] Therefore, as described above, used it, when distributing particles in alkylene glycol, such as ethylene glycol, and it had a suitable distributed function, and it was anxious for development of the dispersing agent which has a desirable good function of an organic component and compatibility. A publicly known dispersing agent which is used for calcium carbonate for paper making which was described above for such the purpose, For example, when the solution of sodium polyacrylate, etc. are used, danger of condensation of the paints particles called a "paints shock" cannot be avoided, The particles by which distributed preparation was carried out will turn into a degree with a condensation coarse particle in an instant very much using synthetic advanced art and grinding classification art, and business may not be made as blocking prevention material, such as polyester film.

[0007]

[Problem(s) to be Solved by the Invention] In view of the above-mentioned actual condition, when this invention persons distribute particles in alkylene glycol, such as ethylene glycol, they have a suitable distributed function, and. About the dispersing agent which has a desirable good function of an organic component and compatibility, wholeheartedly, as a solvent, the dispersing agent using the thing which made alkylene glycol, such as ethylene glycol, contain found out having the very good characteristic, and completed this invention as a result of examination.

[0008] Therefore, the purpose of this invention in alkylene glycol, such as ethylene glycol, Use it, when distributing the particles of silica, kaolin, talc, a titanium dioxide, calcium carbonate, barium sulfate, and others, and it has a suitable distributed function, and is in providing the dispersing agent which has a desirable good function of an organic component and compatibility.

[0009]

[Means for Solving the Problem] This invention is an alkylene glycol content dispersing agent which dissolves a polymer and/or a copolymer which have a specific presentation in a solvent containing alkylene glycol.

[0010] Namely, a dispersing agent which dissolves a polymer in which the 1st of this invention makes alpha, beta mono- ethylenic unsaturated carboxylic acid, or its salt a main composition monomer in a solvent containing alkylene glycol, And the 2nd of this invention makes the contents inorganic particulate dispersion liquid which distributes inorganic particles with the above-mentioned dispersing agent, respectively.

[0011] alpha and beta unsaturated dicarboxylic acid which are chosen from alpha in this invention chosen from acrylic acid, methacrylic acid, crotonic acid, etc. as alpha and beta mono- ethylenic unsaturated carboxylic acid, beta unsaturation monocarboxylic acid, maleic acid, itaconic acid, boletic acid, etc. can be mentioned.

[0012] A polymer in this invention is a polymer which makes alpha, beta mono- ethylenic unsaturated carboxylic acid, or its salt a main composition monomer, One sort or two sorts or more of homopolymers or copolymers of alpha, beta mono- ethylenic unsaturated carboxylic acid, or its salt, And a copolymer of alpha, beta mono- ethylenic unsaturated carboxylic acid or its salt and they, and a monomer that has copolymeric is mentioned, and the following can be mentioned as alpha, beta mono- ethylenic unsaturated carboxylic acid or its salt, and a monomer that has copolymeric.;

(a) Methyl acrylate, ethyl acrylate, acrylic acid propyl, Isobutyl acrylate, 2-ethylhexyl acrylate, methyl methacrylate, Ethyl methacrylate, methacrylic acid propyl, methacrylic acid isobutyl, The acrylic-acid-alkyl-ester and methacrylic acid alkyl ester (b) methoxy ethyl acrylate, such as 2-ethylhexyl methacrylate, Acrylate which has alkoxy groups, such as methoxy ethyl methacrylate and ethoxyethyl acrylate and ethoxyethyl methacrylate, and the methacrylate (c) cyclohexyl acrylate, Acrylate which has cyclohexyl groups, such as cyclohexyl methacrylate, and methacrylate (d) 2-hydroxyethyl acrylate, alpha, such as 2-hydroxyethyl methacrylate, the beta mono- ethylenic unsaturation hydroxy ester (e) polyethylene-glycol monoacrylate, Polyethylene glycol monomethacrylate, PORIPIRO pyrene glycol monoacrylate,

PORIPROPIREN glycol mono- methacrylate, polyethylene-glycol PORIPROPIREN glycol mono- methacrylate, Polyethylene-glycol polytetramethylene glycol mono- methacrylate, Polyalkylene glycol monoacrylate and mono- methacrylate (f) vinyl acetate, such as methoxy polyethylene glycol monomethacrylate, Vinyl system aromatic (h) acrylonitrile, such as vinyl ester (g) styrene, such as stearic acid vinyl, and vinyltoluene, Unsaturated nitrile (i) maleic acid monomethyl, such as a methacrylonitrile, Conjugated dienes, such as vinyl ether (k) butadiene, such as the unsaturated-dicarboxylic-acid ester (j) methyl vinyl ether, such as dibutyl itaconate, and butylvinyl ether, ethylene, propylene, n-butene, isobutene, and n-pentene, an olefin.

[0013]In a copolymer of at least one sort chosen from alpha, beta mono- ethylenic unsaturated carboxylic acid, or its salt, and this alpha, beta mono- ethylenic unsaturated carboxylic acid or its salt and a monomer of others which have copolymeric, 20 % of the weight or more is desirable especially preferred, and a rate of closing to a total monomer of this alpha, beta mono- ethylenic unsaturated carboxylic acid, or its salt is 40 % of the weight or more. When quantity of a monomer of others to which quantity of alpha, beta mono- ethylenic unsaturated carboxylic acid, or its salt has less than 20 % of the weight, i.e., these, and copolymeric exceeds 80 % of the weight, there is a possibility that the dispersibility of particles may become insufficient.

[0014]Although polyalkylene glycol monoacrylate or mono- methacrylate is preferred and is similarly used at 80 or less % of the weight as the above-mentioned alpha, beta mono- ethylenic unsaturated carboxylic acid or its salt, and a monomer that has copolymeric, In order to fully demonstrate an effect of the monomer concerned, it is preferred to use 50 or less % of the weight 5% of the weight or more.

[0015]As for a carboxyl group which exists in polymer or a copolymer, it is preferred that it is perfect neutralizing salt or partial neutralizing salt from a viewpoint of a dispersibility improvement of particles by an alkaline metal, ammonium, amine, etc.

[0016]Further again a molecular weight of a polymer or a copolymer which constitutes this invention, If it is preferred that it is 50000 or less and it says with viscosity from a viewpoint of a dispersion effect of particles, 5000 cps or less will be desirable especially preferred, and viscosity of 40-% of the weight solution will be a thing of 3000 cps or less (a Brookfield viscometer, 25 **, rotor number of rotations of 60 rpm).

[0017]As alkylene glycol in this invention, For example, ethylene glycol, propylene glycol, a butylene glycol, A trimethylene glycol, tetramethylene glycol, pentamethylene glycol, Hexamethylene glycol, a polymethylene glycol of the carbon numbers 2-10 like decamethyleneglycol **, or alicycle fellows diol like cyclohexane dimethanol can be mentioned, and these are independent, or are combined two or more sorts, and are used. From a viewpoint in which an alkylene glycol content dispersing agent of this invention does not spoil water solubility, with a carbon numbers [, such as ethylene glycol, propylene glycol, and a butylene glycol,] of four or less alkylene glycol is preferred, and ethylene glycol is especially the most preferred.

[0018]As for a rate of alkylene glycol over polymer 100 weight section in a dispersing agent of this invention, it is preferred that they are one or more weight sections, and it is still more preferred. [of ten or more weight sections] In the case of less than one weight section, a dispersion effect of particles suspended in alkylene glycol may become insufficient, When exceeding 150 weight sections, since it will be necessary to use a dispersing agent of this invention so much for fully distributing particles and particulate concentration in alkylene glycol dispersion liquid of particles will fall substantially as a result, it is not desirable.

[0019]A dispersing agent of this invention can be easily prepared by using a polymerization method of the usual vinyl system monomer. An ingredient which is a polymerization nature monomer which will become a constituent of a polymer used for this inventions, such as alpha and beta mono- ethylenic unsaturated carboxylic acid, if an example of the preparing method is given, A total monomer is received in the usual polymerization initiator, for example, water soluble persulfate like potassium persulfate, For example, a solvent which contains alkylene glycol, such as ethylene glycol, using 0.1 to 10 % of the weight, For example, water, a mixed solvent of ethylene glycol, or a mixed solvent of alcohol like ethanol, and ethylene glycol, Or it is made to polymerize at about 50-150 ** in a mixed solvent of water, ethanol, and ethylene glycol for about 1 to 10 hours, It can prepare easily by making it distill under decompression of a part of water and ethanol if needed after the end of a polymerization, and adjusting pH with alkali metal salt or an ammonia solution suitably if needed after that.

[0020>About an addition stage of alkylene glycol, As shown in the above-mentioned preparing method, it may add to a solvent before a polymerization start, but a polymerization reaction is performed without adding alkylene glycol to a solvent before a polymerization start, and even if it carries out adding after mixing of the alkylene glycol, such as ethylene glycol after the end of a polymerization reaction, it does not

interfere. It may dissolve in alkylene glycol and powder which polymerizes the above-mentioned polymerization nature monomer may be prepared again.

[0021]
[Example]Although an example is given and this invention is explained in more detail hereafter, this invention does not receive restrictions at all by these. Measurement calculation of the measurement of the particle diameter by the light transmission type particle-size-distribution measuring apparatus in this example is done in the following way.
measuring apparatus kind: -- Shimadzu make SA-CP3 measuring method: -- solvent: -- it shall be as the example computation below solution preliminary distribution:ultrasonic dispersion 100-second measurement temperature:25.0 ** **2.5 ** instrumentation method: which 0.004 % of the weight of sodium polyacrylate dissolved in ion exchange water.

粒度分布測定結果（一例）

粒径（μm）	重量累計%
8. 0 0～6. 0 0	0. 0
8. 0 0～5. 0 0	1. 0
8. 0 0～4. 0 0	3. 0
8. 0 0～3. 0 0	6. 0
8. 0 0～2. 0 0	11. 0
8. 0 0～1. 0 0	18. 0
8. 0 0～0. 8 0	28. 0
8. 0 0～0. 6 0	42. 0
8. 0 0～0. 5 0	58. 0
8. 0 0～0. 4 0	72. 0
8. 0 0～0. 3 0	82. 0
8. 0 0～0. 2 0	89. 0
8. 0 0～0. 1 5	94. 0
8. 0 0～0. 1 0	97. 0
8. 0 0～0. 0 8	99. 0
8. 0 0～0. 0 6	100. 0
8. 0 0～0. 0 0	100. 0

DP1: Particle diameter at the time of a total of 25% of the weight which measured from the big particle diameter side in the particle size distribution measured using light transmission type particle-size-distribution measuring apparatus (Shimadzu SA-CP3) (micrometer)

DP2: Particle diameter at the time of a total of 50% of the weight which measured from the big particle diameter side in the particle size distribution measured using light transmission type particle-size-distribution measuring apparatus (Shimadzu SA-CP3) (micrometer)

DP3: Particle diameter at the time of a total of 75% of the weight which measured from the big particle diameter side in the particle size distribution measured using light transmission type particle-size-distribution measuring apparatus (Shimadzu SA-CP3) (micrometer)

DP 1, 2, and 3 calculated from the above-mentioned particle-size-distribution measurement result becomes as follows.

$$DP1=0.80+(28.0-25.0) \times (1.00-0.80) / (28.0-18.0) =0.86$$
$$DP2=0.50+(58.0-50.0) \times (0.60-0.50) / (58.0-42.0) =0.55$$
$$DP3=0.30+(82.0-75.0) \times (0.40-0.30) / (82.0-72.0) =0.37$$

[0022]The inorganic particles (synthetic spherical silica) used into reference example this example were prepared in the following way. dissolving NaOH0.006g in the water 81.0g -- silica -- 2.3g of sol was added and the mixed stock obtained by adding and stirring 100 g of ethanol further was maintained at 35 **. The mixed liquor of 600 g of ethanol and the water 540g and the 28% ethyl silicate 320g were simultaneously added gradually over 16 hours, having introduced ammonia gas into this mixed stock, and controlling pH to 11.5. The particles obtained from the scanning electron microscope photograph measurement result were spherical silica which has the particle diameter of 0.2 micrometer.

[0023]Acrylic acid 100 weight section was used as example 1 monomer, ammonium-persulfate 3 weight

section was used as a polymerization initiator, and acrylic acid was polymerized with the conventional method as a solvent in the mixed solvent of ethanol 70 weight section, propylene glycol 79 weight section, and water 32.2 weight section. Polymerization temperature trickled 80 **, acrylic acid, and an ammonium persulfate into the solvent in about 5 hours. Almost all ethanol was removed after the end of a polymerization, NaOH-aqueous-solution 115.8 weight section was added 48% of the weight as a neutralizer, pH was prepared to 7.5, and the dispersing agent which consists of a polymer solution of sodium polyacrylate was obtained. The physical properties of the alkylene glycol content dispersing agent obtained by this example are shown in Table 2.

[0024]Two to example 5 monomer, a polymerization initiator, a solvent, a neutralizer, and polymerization temperature were made into the conditions shown in Table 1, and also the polymerization reaction was performed according to Example 1. The physical properties of the alkylene glycol content dispersing agent obtained by this example are shown in Table 2.

[0025]The polymerization reaction was performed without adding propylene glycol to the solvent before a polymerization start among the conditions of example 6 Example 1, and the dispersing agent which consists of a polymer solution of sodium polyacrylate on conditions like Example 1 was obtained to the polymer solution after NaOH addition except having carried out adding after mixing mixing of the propylene glycol. The physical properties of the alkylene glycol content dispersing agent obtained by this example are shown in Table 2.

[0026]One to comparative example 4 monomer, a polymerization initiator, a solvent, a neutralizer, and polymerization temperature were made into the conditions shown in Table 3, and also the polymerization reaction was performed according to Example 1. The physical properties of the dispersing agent which does not contain the alkylene glycol obtained by this comparative example are shown in Table 3.

[0027]

[Table 1]

	実施例 1	実施例 2	実施例 3	実施例 4	実施例 5	実施例 6
組成	AA:100	AA:72 MAA:28	AA:20 ITA:10 MA:70 チオ : 2	AA:90 AN:2 VAC:2 St:5 PGMMA:1 チオ:1	AA:60 PGMMA:40	実施例 1 と同じ。 但し P G は、NaOH 添加後、 後添加し 混合した
開始剤	APS:3	AIBN:1	AIBN:1	APS:1	APS:3	
溶媒	H ₂ O:32.2 EtOH:70 PG:79	IPA:100 DEG: 140.2	EG:143.5	H ₂ O: 169.5 MeOH:100 EG:5	H ₂ O:32.2 IPA:70 EG:80	
中和剤	48%NaOH: 115.8	48%NaOH: 36.8	25%NH ₄ OH :29.3	48%NaOH: 52	25%NH ₄ OH :12	48%NaOH: 115.8
温度 (℃)	80	85	90	70	85	80

[0028]

[Table 2]

実施例	1	2	3	4	5	6
濃度 (%)	40	40	40	40	40	40
pH	7.5	2.5	7.5	7.5	4.5	7.5
粘度(cps)	195	2500	410	1530	220	305

[0029]

[Table 3]

	比較例 1	比較例 2	比較例 3	比較例 4
組成	AA:100	AA:72 MAA:28	AA:20 ITA:10 MA:70 チオ : 2	AA:90 AN:2 VAC:2 St:5 PGMMA:1 チオ : 1
開始剤	APS:3	AIBN:1	AIBN:1	APS:1

溶媒	H ₂ O:111.2 EtOH:70	IPA:100 H ₂ O:140.2	H ₂ O:143.5	H ₂ O:174.5 MeOH:100
中和剤	48%NaOH: 115.8	48%NaOH: 36.8	25%NH ₄ OH :29.3	48%NaOH: 52
重合温度 (°C)	80	85	90	70
濃度 (%)	40	40	40	40
pH	7.5	2.5	7.5	7.5
粘度 (cps)	250	2800	400	2100

All the numbers the units in the monomer composition of Table 1 and Table 3, a polymerization initiator, a solvent, and a neutralizer are not indicated to be express the weight section. The details of the cable address in Table 1 and Table 3 are shown below.

AA [... Methyl acrylate AN / ... Acrylonitrile, PGMMA / ... Polyethylene glycol monomethacrylate (molecular weight: about 400)] ... Acrylic acid and MMA ... Methacrylic acid, ITA ... Itaconic acid and MA
St ... Styrene and VAC ... Vinyl acetate APS ... Persulfuric acid Ain More, azobisisobutironitoriru ...
Azobisisobutyronitrile EtOH ... Ethyl alcohol, H₂O [... Propylene glycol,] ... Water IPA ... Isopropyl alcohol,
MeOH ... Methyl alcohol PG EG [.. Ammonium hydroxide thio / ... Regulator viscosity / ... Measured value
by 40% of concentration, 25 **, and a Brookfield viscometer] ... Ethylene glycol DEG ... A diethylene glycol,
NaOH ... Sodium hydroxide NH₄ OH [0030]To the mixed stock of the spherical silica, ethanol, and water
which were obtained by the example 7 aforementioned reference example. As a polymer the alkylene glycol
content dispersing agent obtained in Example 1 Per spherical silica solid content, Added 3.0% of the weight,

supplied ethylene glycol after stirring, carried out Flushing using the rotating evaporator, water and ethanol were made to remove from this mixed stock, and the survival rate of water obtained the ethylene glycol dispersion liquid whose solids concentration of spherical silica is 20.0 % of the weight 0.4%. The particle-size-distribution measurement result of the ethylene glycol dispersion liquid of the spherical silica obtained by this example 7 is shown in Table 4. The particle-size-distribution measurement result of Table 4 shows that the ethylene glycol dispersion liquid of the spherical silica obtained by this example 7 have very good dispersibility.

[0031]The ethylene glycol dispersion liquid whose solids concentration of spherical silica the alkylene glycol content dispersing agent used for eight to example 12 Example 7 was changed into the alkylene glycol content dispersing agent shown in Table 4, and also is 20 % of the weight similarly were obtained. The particle-size-distribution measurement result of the ethylene glycol dispersion liquid of the spherical silica obtained by this examples 8-12 is shown in Table 4. The particle-size-distribution measurement result of Table 4 shows that the ethylene glycol dispersion liquid of the spherical silica obtained by this examples 8-12 have very good dispersibility.

[0032]The ethylene glycol dispersion liquid whose solids concentration of spherical silica the alkylene glycol content dispersing agent used for five to comparative example 8 Example 7 was changed into the alkylene glycol content dispersing agent shown in Table 4, and also is 20 % of the weight similarly were obtained. The particle-size-distribution measurement result of the ethylene glycol dispersion liquid of the spherical silica obtained by these comparative examples 5-8 is shown in Table 4. From the particle-size-distribution measurement result of Table 4, the ethylene glycol dispersion liquid of the spherical silica obtained by these comparative examples 5-8 contained the aggregate particle, and dispersibility was poor.

[0033]

[Table 4]

	使用した分散剤	粒度分布測定結果			
		DP 1	DP 2	DP 3	DP 1 / DP 3
実施例 7	実施例 1 調製品	0.24	0.21	0.17	1.412
実施例 8	実施例 2 調製品	0.24	0.21	0.16	1.500
実施例 9	実施例 3 調製品	0.23	0.20	0.17	1.353
実施例 10	実施例 4 調製品	0.22	0.20	0.18	1.222
実施例 11	実施例 5 調製品	0.22	0.20	0.19	1.158
実施例 12	実施例 6 調製品	0.24	0.21	0.16	1.500
比較例 5	比較例 1 調製品	0.59	0.45	0.20	2.950
比較例 6	比較例 2 調製品	0.54	0.36	0.28	1.929
比較例 7	比較例 3 調製品	0.88	0.54	0.21	4.190
比較例 8	比較例 4 調製品	0.95	0.66	0.32	2.969

[0034]The ethylene glycol dispersion liquid of spherical silica prepared by the dispersibility test examples 7, 8, 9, 10, 11, and 12 and the comparative examples 5, 6, 7, and 8 were further diluted with ethylene glycol, and the diluent was prepared each 10% of the weight. A 0.8-micrometer membrane filter (made by Millipore Corp.) was filtered under pressure on condition of 2 kg/cm² immediately after preparation of these 10-% of the weight diluents and after 24-hour settlement, and the through put was measured. A measurement result is shown in Table 5. From the result of Table 5, the ethylene glycol dispersion liquid of spherical silica prepared in each example, As compared with the ethylene glycol dispersion liquid of spherical silica prepared by the comparative example, filtering nature is good, and blinding of the filter by secondary condensation coarse particle generating in temporality does not have it, either, and it is checked that dispersibility with the passage of time is good.

[0035]

[Table 5]

	調製直後の通過量 (ml)	24時間後通過量 (ml)
実施例7の10wt%希釈液200ml	200	200
実施例8の10wt%希釈液200ml	200	200
実施例9の10wt%希釈液200ml	200	200
実施例10の10wt%希釈液200ml	200	200
実施例11の10wt%希釈液200ml	200	200
実施例12の10wt%希釈液200ml	200	200
比較例5の10wt%希釈液200ml	100	80
比較例6の10wt%希釈液200ml	110	60
比較例7の10wt%希釈液200ml	110	60
比較例8の10wt%希釈液200ml	80	20

[0036]After carrying out the ester interchange of application dimethyl terephthalate 100 weight section and the ethylene glycol 70 weight section as a conventional method by making 0.035 copy of manganese acetate 4 hydrate into a catalyst, The ethylene glycol dispersion liquid of spherical silica prepared in Example 7 were added under stirring so that spherical silica concentration might be set to 4000 ppm to polymer. Then, the polycondensation reaction was performed under the elevated-temperature vacuum as the conventional method, and polyethylene terephthalate of limiting viscosity 0.630 was obtained. After extending to the lengthwise direction at dissolution extrusion and 90 °C and extending this polymer 3.5 times in a transverse direction at 3.5 times and 130 °C at 290 °C, it heat-treated at 220 °C and the 15-micrometer-thick film was created. This film was measured according to ASTM-D01894-63T. The maximum surface roughness of the obtained film was 0.106 micrometer.

[0037]The ethylene glycol dispersion liquid of spherical silica prepared by the comparison application comparative example 5 were used, and also it was operated like the application. The maximum surface roughness of the obtained film is 0.250 micrometer, and a big and rough projection is conspicuous.

[Translation done.]